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# Effects of thermomechanical processing on the recrystallization texture and grain size of Al-1%Si sputtering target material

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**Abstract.** An Al-1%Si alloy was solution treated and deformed by conventional cold rolling to different strains, followed by annealing at various temperatures until complete recrystallization. The microstructures of annealed samples were characterized by electron backscatter diffraction. It is found that under optimal conditions of cold rolling and annealing, the microstructure desired for sputtering target materials with fine, uniformly sized and randomly textured grains can be obtained for the Al-1%Si alloy.

## 1. Introduction

Al-Si alloys are widely used as sputtering target materials in semiconductor industry for deposition of thin films [1, 2]. The key factors influencing the properties of sputtered films are the crystallographic texture and grain size. In general, a weak texture and a fine and uniform grain structure are desired for sputtering target materials. It has been well established that grain refinement in metals can be achieved by severe plastic deformation and such an approach is applicable for sputtering target materials. For example, severe plastic deformation by equal channel angular extrusion has been applied to produce sputtering targets [3]. However, this method does not align grains in a manner that facilitates uniform sputtering [4, 5]. Fine grain sizes were also obtained by deforming the materials at very low temperatures [6, 7]. It is more challenging to produce a weak or random texture in sputtering target materials. Plastic deformation in a complicated manner, i.e. clock rolling, has been developed to obtain weak textures for sputtering targets [8]. In this study, we report that Al-1%Si target with a random texture, and fine and uniform grain structure and can be produced by thermomechanical processing involving solid solution treatment, cold rolling to high strains and recrystallization annealing.

## Experiment

An Al-1%Si alloy was casted from ultra-high purity (99.9996%) Al and high purity Si. The ingot was deformed at about 200°C into a thick slab, which had a mean grain size of about 150 µm. A plate with dimensions of 50 × 100 × 20 mm<sup>3</sup> was cut from the slab, and solid solution treated at 540°C for 6 hours followed by water quenching. Then the plate was cold



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rolled at room temperature to 63, 86, 95 and 98% reductions in thickness, respectively. These samples were annealed at 250, 350 and 450°C for different times until complete recrystallization. The microstructures of the samples were observed in a Zeiss Auriga dual beam station, and local crystallographic orientations were characterized using an Oxford HKL Channel 5 electron backscatter diffraction (EBSD) detector attached to the microscope. For EBSD measurements, the specimen surface was mechanically polished followed by electrochemical polishing in the A2 solution. A step size of 2  $\mu\text{m}$  was used in EBSD scanning. The grain size was determined by the equal circular diameter method of the Channel software with a 5° critical angle in the data processing.

## Results

The distribution of Si particles after solid solution treatment and recrystallization annealing at 350°C for 6 h are shown in Fig. 1(a) and Fig. 1 (b), respectively. It is seen that after solid solution treatment at 540°C for 6 hours, majority of Si particles have been dissolved. However, a small amount of coarse particles or particle clusters are retained, distributing along the rolling direction (RD) of the raw plate. After recrystallization annealing at 350°C for 6 h, fine nano-sized Si particles precipitate and fairly randomly distribute in the matrix. There are some large particles of a few micrometers. These large particles could be formed by ripening of the retained particles during annealing.

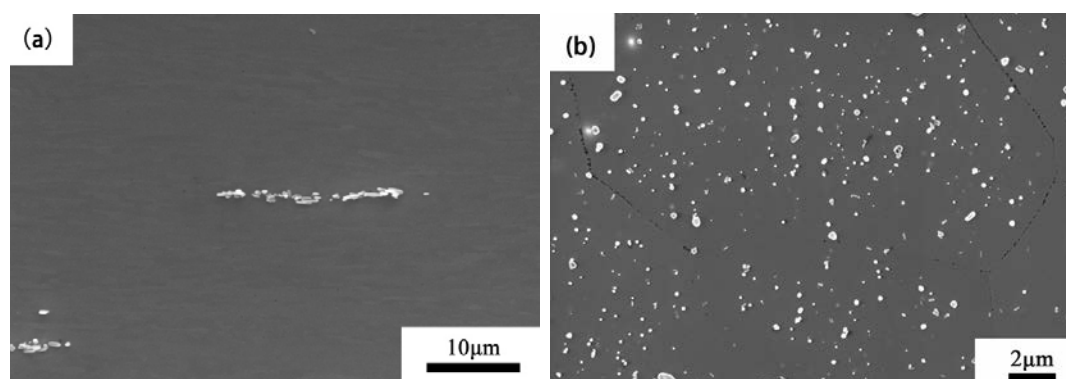


Fig. 1 SEM micrographs of Si particles. (a) after solid solution treatment and (b) after cold rolling to 86% and annealing at 350°C.

Figure 2a shows the average grain size of all treated samples after complete recrystallization. A general observation is that the samples annealed at 450°C show larger average grain sizes than the samples annealed at 250°C. For both annealing temperatures of 250 and 450 °C, the average grain size decreases with increasing deformation strain, except for the case of 98% deformed sample annealed at 450°C. However, for the annealing at 350°C, the evolution shows a more complicated pattern with the 86% deformed sample showing the finest grain size after annealing. Another general observation is that with increasing strain, the grain structure becomes more homogeneous, as shown by the standard deviation of grain size distribution in Fig. 2b. The 63% deformed sample has the largest spread of grain sizes (20-40  $\mu\text{m}$ ), whereas the 98% deformed sample the smallest (15-20  $\mu\text{m}$ ). As for the recrystallization texture, a rather weak (random) texture was developed in all recrystallized samples. The 250°C annealed samples have ~9-23% rolling texture components, while 350°C and 450°C annealed samples only have less than 10% rolling texture components. All samples have ~3-5% of cube texture.

It is found that the sample deformed to 86% and annealed at 350°C for 6h has the smallest grain size and a random texture. Figure 3 (a) shows the EBSD map of the sample. It is clear

that the material is completely recrystallized, and the grains are fine and equiaxed. The recrystallization texture of Al-1%Si is shown in Fig. 3 (b). It can be seen from the orientation distribution function (ODF) map that the recrystallized texture of the alloy is relatively random. Beside weak textures around cube  $\{100\}<001>$ , rotated cube  $\{100\}<011>$  (R-cube) and rotated Goss  $\{110\}<110>$  (R-Goss), other are random orientations.

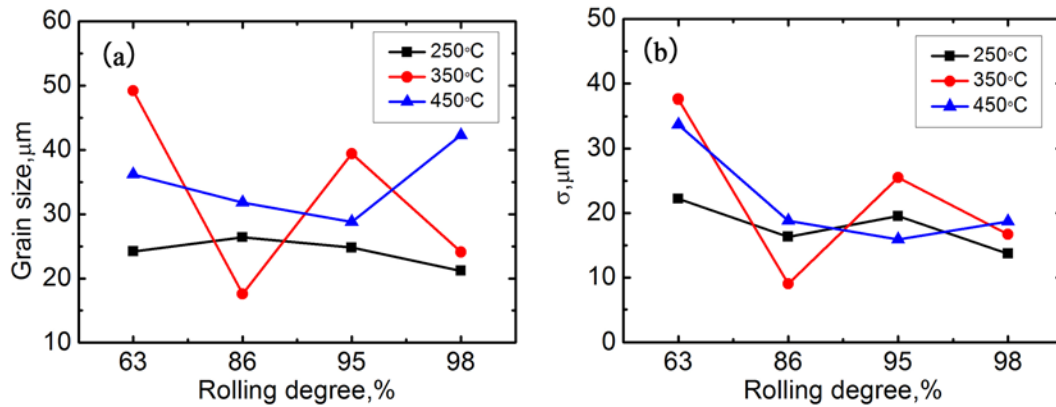


Fig. 2. (a) Average grain size and (b) standard deviation of grain size distribution of Al-1%Si after complete recrystallization.

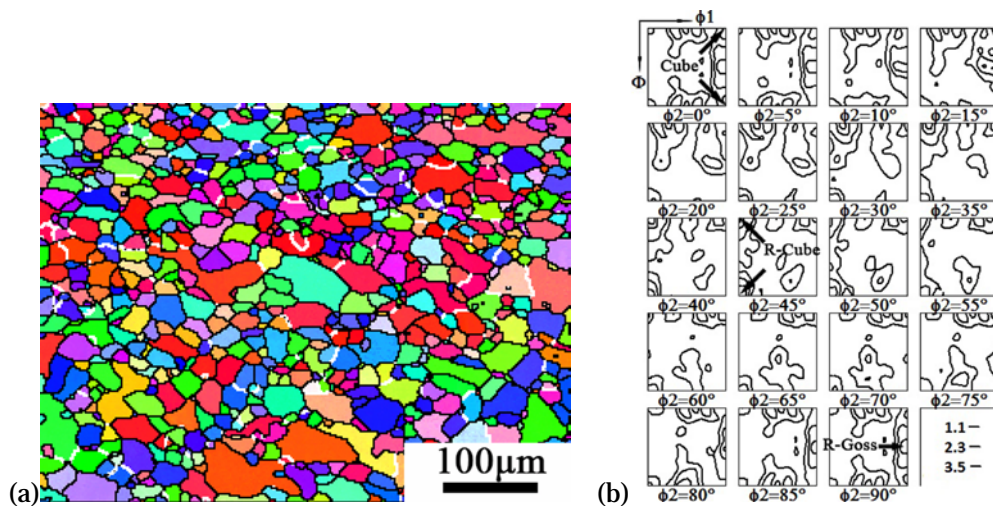


Fig. 3 (a)EBSD map and (b) ODF of the sample deformed to 86% and annealed at 350°C. On the EBSD map, black and white lines represent boundaries of misorientation  $\geq 15^\circ$  and  $2-15^\circ$ , respectively.

Table 1 shows the orientation content ratio (represented by the percentage of grains with their normal parallel to a specific crystallographic direction [7]) of the sample deformed to 86% and annealed at 350°C for 6h. It is seen that the percentages of  $\{200\}$  and  $\{220\}$  orientations are about 20-25%, and the  $\{311\}$  orientations account for about 31%, while the  $\{111\}$  orientations are greatly suppressed. The combination of a weighted  $\{200\}$  orientation and balanced  $\{111\}$ ,  $\{220\}$  and  $\{311\}$  orientations is expected to give uniform sputter properties from the sputter target [7] made from the present alloy.

Table 1 Orientation content ratio of the sample deformed to 86% annealed at 350°C

	$\{111\}$	$\{200\}$	$\{220\}$	$\{311\}$
Ratio, %	9	26	21	31

## Discussion

Usually the average recrystallization grain size decreases with increasing deformation strain since the stored energy increases with strain and thus promotes recrystallization nucleation, which results in a reduced average grain size after recrystallization. For the present alloy, the Si element was present in the solid solution before deformation. The over-saturated Si atoms precipitate during annealing concurrent with the recrystallization of the Al matrix. Precipitated fine particles can pin the migration of grain boundaries during recrystallization due to the Zener-drag effect. Therefore the grain size is mainly controlled by the distribution and size of Si precipitates. For the samples annealed at 250°C, the Si precipitates are all fine and randomly distributed. So the average grain sizes are generally small. For the samples annealed at 450°C, precipitates are coarsened at the temperature, so the grain size are relative large. Especially for the sample deformed to 98% and annealed at 450°C, precipitations are ripped to large sizes so that the pinning effect of particles is rather weak. Very coarse grain sizes are obtained in the sample after recrystallization. For the samples annealed at 350°C, the large grain size may be related to the short annealing time so that the precipitations are not sufficient and extensive growth occurred. As the case of the sample deformed to 86% and annealed at 350°C, Si particles are dispersed and uniformly distributed (Fig 1b) and thus a small grain size is achieved (Fig. 3a).

A texture dominated by random orientations is developed in this alloy after recrystallization. The volume fractions of rolling texture components are very small. This manifests that nucleation from deformation microstructure with rolling texture components is very limited. The reason for the formation of the random texture could be the particle stimulated nucleation since there are large sized particles in the materials before deformation (Fig. 1a) [9, 10]. Another reason could be the confined growth of recrystallizing grains during annealing due to the presence of fine Si particles. The growth advantage of grains with specific orientations, i.e. cube, will be inhibited. Therefore all grains may grow at similar rates, leading to a random texture.

## Conclusion

An Al-1%Si alloy was solid solution treated followed by cold rolling and annealing. The microstructural and textural evolutions were characterized by EBSD. Most of Si element has been dissolved after solution treatment and fine Si precipitates form during recrystallization annealing. Fine and uniform grains as well as a random recrystallization texture are developed in the alloy deformed to 86% followed by complete recrystallization at 350°C.

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